filled layer is filled with 40-75 weight %, based on th total weight of the filled layer, of an inorganic filler, forms one of the two surface layers of the film, and has a thickness of 40 μm to 400 μm, the unfilled layers comprising at least a barrier layer and a sealing layer and optionally at least one adhesive layer, and the ratio of the total thickness of the unfilled layers to the thickness of the filled layer being from 1:8 to 1:1.2.

REMARKS

Claims 1-17 are presently pending in the application.

The Examiner has objected to the incorporation by reference of industrial test standard ASTM 1238. The Examiner asserts that reference to industrial test standard ASTM 1238 is improper because the disclosure allegedly does not enable one of ordinary skill in the art to duplicate the procedure associated with the industrial test. Although standard industrial tests promulgated by the American Society of Testing and Materials are well known in the art and readily available, applicants have submitted herewith a copy of ASTM 1238 from the year 1990 as suggested by the Examiner. Accordingly, reconsideration and withdrawal of this objection are respectfully requested.

The Examiner has objected to claims 1-17 because of informalities. The Examiner argues that the term "of³" in line 4 of claim 1 is not clear. Claim 1 has been amended to delete the superscript numeral "3". The superscript "3" was a typographical error originally intended to be the symbol "≥". Rather than insert the symbol "≥", claim 1 has been amended to specify that the thickness of the filled layer is 40 μm to 400 μm. Support for this amendment is found at least at page 3, lines 24-25 of the specification. Additionally, claim 1 has been amended to substitute the more commonly used term "ratio" for "relation." Accordingly, reconsideration and withdrawal of this objection are respectfully requested.

The Examiner has rejected claims 1-17 under 35 U.S.C. § 112, second paragraph, as being indefinite, asserting that the term "paper-like" is indefinite because it has no accepted meaning in the art and it is unclear how the laminate is like paper. Although it is believed that the term "paper-like" could be readily understood by one skilled in the art, claim 1 has been amended to clarify that the barrier film has the appearance and texture of paper. Support for this

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amendment is found at least at page 8, lines 6-7 of the specification. Accordingly, reconsideration and withdrawal of this rejection are respectfully requested.

The Examiner has further rejected claim 17 under 35 U.S.C. § 112, second paragraph, as being indefinite, arguing that the term "SiO_x" is indefinite because it is not clear from the disclosure what "x" represents. The applicants respectfully traverse this rejection. One of ordinarily skill in the art would readily recognize that silicon oxides are used as inorganic fillers in laminates and films (see e.g., col. 4, line 14 of U.S. Patent No. 4,567,089 of Hattori *et al.*) and that SiO_x refers to the entire class of silicon oxides, for example silicon monoxide when x=1 and silicon dioxide when x=2. Accordingly, reconsideration and withdrawal of this rejection are respectfully requested.

The Examiner has rejected claims 1-4, 6, 7, 10, and 11 under 35 U.S.C. § 103(a) as being unpatentable over U.S. Patent No. 4,526,823 of Farrell *et al.* ("Farrell") in view of U.S. Patent No. 4,567,089 of Hattori *et al.* ("Hattori"). The Examiner argues that Farrell teaches a plastic laminate sheet that comprises an outer layer of filled-plastic, an inner layer of HDPE (high density polyethylene), and an EVOH (ethylene vinyl alcohol) barrier layer interposed between the inner and outer layers. The EVOH barrier layer adheres to the inner and outer layers by an adhesive such as copolymers of olefins and acid or copolymers of ethylene and vinyl ester. The Examiner asserts that Farrell teaches blending 5 to about 80 percent by weight of filler, such as calcium carbonate, talc, or mica into the outer layer. The Examiner additionally asserts that the laminate may be thermo-formed.

The Examiner concedes that Farrell does not teach the claimed thickness ratio of filled layer to unfilled layers. However, the Examiner asserts that Hattori teaches a thermo-formable laminate comprising a filled polypropylene layer and that sufficient deep draw properties are obtained when the filled polymer sheet comprises 55-99.85% of the laminate's total thickness. The Examiner contends that, accordingly, it would have been obvious to a skilled artisan to vary the thickness ratio of the filled layer to the unfilled layers in order to obtain good deep drawing properties. The Examiner further argues that applicant shows no unexpected results relating to the variation of the film thicknesses. This rejection is respectfully but strenuously traversed for the reasons set forth in detail below.

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Farrell teaches away from adjusting the thickness ratio of unfilled layers to filled layers to obtain a laminate with better thermo-formability characteristics, such as deep drawing qualities, by suggesting that "the thickness of each layer is not per se critical [when forming the laminate sheet]." (col. 4, lines 60-63). Further, Farrell suggests a maximum ratio of 1:1 for the laminate (see Examples I and II at col. 5), which is not encompassed by the 1:8 to 1:1.2 range of ratios of the claimed invention. Additionally, Farrell does not recognize the significance of the thickness ratio of filled layer to unfilled layers on the properties of the laminate. As discussed, below applicants provide data showing that the thickness ratio of the filled to unfilled layers provides better thermo-forming characteristics, such as packaging speed and thermo-formability over a broad range of temperatures.

Hattori in no way makes up for the deficiencies of Farrell discussed above. First, a person of ordinary skill in the art would not have been motivated to make such a combination. The total thickness of the film disclosed in the Hattori reference is 1.6 mm or 1600 μm (see col. 8, line 3). The thickness of the unfilled surface layer is 0.5-45% of the total thickness of the laminated sheet (see abstract). Accordingly, the thickness of the filled layer is 55-99.5% of the total thickness of the laminate. Thus, the ratio of the thickness of the unfilled layers to that of the filled layer is 1:1.222 to 1:199. A skilled artisan would not have been motivated to employ the broad ratio of Hattori to adjust the thickness ratio of Farrell to the much narrower and critical range of ratios as claimed by the applicants, when Farrell teaches that "the thickness of each layer is not per se critical." The criticality of the range is further supported by the enclosed unsigned Declaration having data showing the resultant improved properties, including packaging speed and thermo-formability over a broad temperatures, as discussed below.

Additionally, the above ratio of Hattori dictates a thickness of the filled layer of Hattori of 880 to 1592 μm, while the thickness of the filled layer of the Farrell reference is 3 to 7 mils or 76.2 to 177.8 μm or (col. 4, lines 60-66). One of ordinary skill of would not have had an expectation of success in combining Hattori having its much thicker filled layer with Farrell having a much thinner filled layer to achieve the claimed invention, as Hattori teaches that a thickness less than its lower limit for the above-specified range decreases the desirable properties of its laminate (see col. 5, lines 42-44).

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Additionally, Hattori teaches away from combining it with other references to change the combination of film layers. Hattori teaches that the thermo-forming properties of the film are influenced by the type of polymers used for the different layers of the film and the combination of the layers made of different polymers (see col. 1 and Table 1). As shown in Table 1 and claim 1, the melt flow rate and the Q value (a ratio of a weight average molecular weight to a number average molecular weight) of the polymers have an important influence of the properties of the film. Hattori does not disclose that the unfilled layers comprise at least a barrier layer. Therefore, a skilled artisan is directed away from combining Hattori with other references to change the combination of the film layers, such as adding a barrier layer, as would result from combining Farrell and Hattori to achieve the claimed invention. Accordingly, reconsideration and withdrawal of this rejection are respectfully requested.

The Examiner has rejected claims 1-4, 6, 7 and 9-15 under 35 U.S.C. § 103(a) as being unpatentable over U.S. Patent No. 5,011,735 of Schirmer *et al.* ("Schirmer") in view of Hattori. The Examiner contends that Schirmer teaches a thermo-forming laminate comprising a surface film that comprises a polypropylene or ethylene-propylene copolymer, a barrier film that comprises an ethylene vinyl alcohol or polyvinylidene chloride, an optional adhesive layer, and a sealant film that comprises a polyolefin. The Examiner asserts that the laminate may be cross-linked to broaden the range of temperatures at which the laminate may be thermoformed. The Examiner additionally argues that the laminate may be thermoformed on a FFS machine and sealed with lidding film.

The Examiner admits that Schirmer does not teach the claimed thickness ratio of filled layer to unfilled layers, but argues that Hattori cures this deficiency as argued with respect to Farrell. The Examiner also acknowledges that Schirmer does not teach that the polypropylene layer may contain 40-75% by weight of inorganic filler, but contends that Hattori teaches a filled polypropylene layer with 5-60% of a filler selected from the group consisting of calcium carbonate, silica, talc, clay, titanium oxide, etc. The Examiner asserts it would have been obvious to one of ordinary skill in the art to add 5-50 parts by weight filler to the propylene layer taught in Schirmer to improve the laminate's heat resistance, stiffness, and dimensional stability. This rejection is respectfully but strenuously traversed for the reasons set forth in detail below.

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Schirmer does not teach or suggest that the relative thickness of the layers is critical to the properties of the film. Hattori similarly does not cure the deficiencies of Schirmer, as a skilled artisan would not have been motivated to make such a combination. As discussed above, a skilled artisan would not have been motivated to employ the broad ratio of Hattori to adjust the thickness ratio of Schirmer to achieve the much narrower and critical range of ratios as claimed by the applicants. Additionally, as discussed above, Hattori directs a skilled artisan away from combining Hattori with other references to change the combination of the film layers, such as adding a barrier layer which Hattori does not disclose, as would result from combining Schirmer and Hattori to achieve the claimed invention.

Further, Schirmer does not teach or suggest any filling of the propylene layer. Hattori in no way makes up for this deficiency. First, there is no motivation or suggestion to make the Examiner's proposed modification with Hattori. Schirmer requires the laminate to be capable of being stretched for vacuum skin packaging or thermo-forming (see col. 3, lines 42-45, col. 2, lines 11-14 and 53-55). Hattori, however, requires a stiffer product suitable for use in the fabrication of food trays, car parts, etc. as substitutes for conventional sheets of polystyrene (see col. 3, lines 48-49 and col. 1, lines 18-22). Thus Schirmer aims to achieve high formability (see title), while Hattori seeks to balance formability with stiffness, two divergent properties (see col. 4, lines 18-20 and 52-57). Accordingly, a skilled artisan would have been directed away from combining Hattori with Schirmer by adding an inorganic filler to improve the stiffness of the Schirmer product as asserted by the Examiner.

Additionally, Schirmer teaches its laminate, in order to increase the thermo-forming temperature range of the laminate, must have one of its layers cross-linked (col. 26-29). Absent such cross-linking, the temperature at which the thermo-forming process may be conducted will range "from only a few degrees to about 10°C." (col. 5, lines 29-33). Accordingly, Schirmer recommends cross-linking if a broader temperature for thermo-forming is desired. A skilled artisan would not have been motivated to combine Hattori with Schirmer to achieve a broader range of temperatures by adding a filler to one of the layers and adjusting the thickness of each layer relative to the other, as accomplished by the presently-claimed film, rather than cross-linking a layer of the laminate as recommended by Schirmer. Accordingly, reconsideration and withdrawal of this rejection are respectfully requested.

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The Examiner has rejected claim 8 under 35 U.S.C. § 103(a) as being unpatentable over Schirmer in view of Hattori, as applied above, further in view of US. Patent No. 4,567,089 of Bochow *et al.* ("Bochow"). The Examiner acknowledges that neither Schirmer nor Hattori teaches that the sealing layer may comprise LDPE or a blend of polybutene and LDPE. The Examiner argues that Bochow teaches a multilayer composite film comprising a filled polypropylene film, an adhesive layer, a gas barrier layer, a second adhesive layer, and heat sealing layer that may comprise LDPE, polybutylene, ethylene vinyl acetate, ethylene acrylic copolymers and blends thereof. The Examiner argues that it would have been obvious to one of ordinary skill in the art to utilize LDPE, or a blend of polybutene and LDPE as the sealing layer since Bochow teaches both compositions are known in the art as good sealing layers in barrier films. This rejection is respectfully but strenuously traversed for the reasons set forth in detail below.

First, the combination of Schirmer and Hattori is improper for the reasons discussed above with regard to claims 1-4, 6, 7 and 9-15. Second, a skilled artisan would not have been motivated to further combine Bochow with the combination of Schirmer and Hattori. Bochow is directed to a white opaque film with a high degree of stiffness and limited thermo-formability. By contrast, both Schirmer and Hattori require improved thermo-formability, and Schirmer additionally aims to achieve from its laminate a clear packaging material with good clarity and gloss (see col. 1, lines 41-43). A skilled artisan would not have been motivated to modify the thermo-formable laminates of Schirmer and Hattori by adding the LDPE or LDPE blend of Bochow which aims to achieve limited thermo-formability. A skilled artisan would also have found no motivation to combine Schirmer which seeks a clear package with good clarity with Bochow which seeks an opaque film. Accordingly, reconsideration and withdrawal of this rejection are respectfully requested.

The Examiner has rejected claims 16 and 17 under 35 U.S.C. § 103(a) as being unpatentable over Schirmer in view of Hattori, further in view of applicants' admissions. The Examiner acknowledges that neither Schirmer nor Hattori teaches that the lidding film may comprise the multilayered films claimed in claims 16 and 17. The Examiner contends that applicant admits in the specification that both claimed lidding films are well known in the art, and so it would have been obvious to one of ordinary skill in the art to utilize either because of

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applicant's admissions. The rejection is respectfully but strenuously traversed for the reasons set forth below.

First, the combination of Schirmer and Hattori is improper for the reasons set forth above. Second, a skilled artisan would not have been motivated to further combine applicants' admissions with the combination of Schirmer and Hattori. As discussed above, Hattori teaches that the thermo-forming properties of the film are influenced by the type of polymers used for the different layers of the film and the combination of the layers made of different polymers (see Table 1). Thus, Hattori directs a skilled artisan away from combining Hattori with other references, including applicants' own disclosure, to encompass undisclosed types of polymer blends as would result from combining Schirmer and Hattori with applicants' statement to achieve the claimed lidding films. Additionally, Schirmer aims to achieve a laminate with high gloss and clarity while applicant achieves a film with the appearance and texture of paper. A skilled artisan would not have been motivated to use the multilayered lidding films as disclosed by the applicants as applicants achieve an entirely different appearance and texture than does Schirmer. Accordingly, reconsideration and withdrawal of this rejection are respectfully requested.

The Examiner has rejected claim 5 under 35 U.S.C. § 103(a) as being unpatentable over Farrell or Schirmer in view of Hattori, as applied above, further in view of U.S. Patent No. 5,635,011 of Rosen ("Rosen"). The Examiner admits that neither Farrell nor Schirmer nor Hattori teaches that the matrix polymer may be adhered without an adhesive to a layer comprising a blend of the matrix polymer with EVOH or PA. The Examiner asserts that Rosen teaches that it is known in the art to blend a matrix resin with a barrier layer in order to eliminate an adhesive layer between two layers of a laminate. The Examiner argues that it would have been obvious to use a blend of matrix polymer with EVOH or PA as the barrier layer of the laminates taught in Farrell or Schirmer in view of Hattori, because it is known in the art that these barrier layers eliminate the need of an adhesive layer. This rejection is respectfully but strenuously traversed for the reasons set forth below.

First, the combination of Farrell or Schirmer with Hattori is improper for the reasons discussed above. Second, there is no motivation to further combine Rosen with the combination of Farrell or Schirmer with Hattori. Rosen is directed to a laminate with a barrier

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layer containing an electrically conductive material in particle form (see col. 3, lines 11-14). Rosen also seeks making its packaging material completely impenetrable to light (see col. 1, lines 35-36), while Schirmer seeks a clear packaging material with good clarity. Accordingly, a skilled artisan would not have been motivated to modify Schirmer which seeks clear packaging material with Rosen which seeks making its packaging material impenetrable to light. As discussed above, Hattori teaches away from altering its combination and types of polymers. Accordingly, a skilled artisan would not have been motivated to look to Rosen for a matrix polymer blend to substitute for the adhesive layer in Hattori. Accordingly, reconsideration and withdrawal of this rejection are respectfully requested.

The Examiner has additionally rejected claim 5 under § 103(a) as being unpatentable over Farrell or Schirmer in view of Hattori, as applied above, further in view of U.S. Patent No. 5,108,844 of Blemburg et al. ("Blemburg"). The Examiner acknowledges that neither Farrell nor Schirmer nor Hattori teaches that the matrix polymer may be adhered without an adhesive layer comprising a blend of matrix polymer with EVOH or PA. The Examiner contends that Blemburg teaches that two layers may be adhered by blending some of each composition into the adjacent layer, and, accordingly, it would have been obvious to one of ordinary skill to use a blend of matrix polymer with EVOH or PA as the barrier laminates, because Blemburg teaches that two layers can be adhered together without the use of an adhesive layer by blending some of each composition into the adjacent layer. This rejection is respectfully but strenuously traversed for the reasons set forth below.

First, Farrell or Schirmer and Hattori are improperly combined as discussed above. Additionally, there is no motivation to further combine Blemburg with Farrell or Schirmer and Hattori. As discussed above, Hattori teaches that the thermo-forming properties of the film are influenced by the type of polymers used for the different layers of the film and the combination of the layers made of different polymers (see Table 1 and claim 1). Thus, Hattori directs a skilled artisan away from combining any combination of Hattori with Blemburg to encompass undisclosed types of polymer blends as would result from combining any combination of Hattori with Blemburg. Additionally, Blemburg suggests that films thicker than 76 μm are less economical and films up to about only 510 μm are functional (see col. 4, lines 37-39). As discussed above, the thickness of the film in Hattori is 1600 μm (see col. 8, line 3).

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Accordingly, a skilled artisan would not have been motivated to look to Blemburg to modify any combination of Hattori when Blemburg suggests that Hattori's film is not functional. Therefore, reconsideration and withdrawal of the rejection are respectfully requested.

The Examiner has rejected claims 1, 2, 4, 6-11, 13 and 14 under 35 U.S.C. § 103(a) as being unpatentable over Bochow in view of Hattori. The Examiner argues that Bochow teaches a multilayer, thermo-formable composite film with a surface layer, an optional adhesive layer, a gas barrier layer, a second optional adhesive layer, and a heat sealable layer. The surface layer comprises a polypropylene matrix resin and filler. The barrier layer comprises polyamide, polyvinyl alcohol, ethylene vinyl alcohol or polyesters. The heat sealing layer comprises a polyolefin or amorphous polyester such as LDPE, polybutylene, EVA, ethylene-carboxylic acid copolymers and mixtures thereof. Bochow discloses specific thicknesses for each of the layers. The Examiner argues that the ranges disclosed overlap the claimed ranges, rendering the claimed ratios obvious.

The Examiner admits that Bochow does not teach how much filler should be added to the film. The Examiner contends that Hattori teaches that the filler should comprise 5-60% of the polypropylene layer. The Examiner asserts that it would have been obvious to one of skill in the art to add 5-50 parts by weight filler to the propylene layer in Bochow to improve the laminate's heat resistance, stiffness, and dimensional stability. Additionally, the Examiner acknowledges that Bochow does not teach that the laminate may be formed on an FFS machine. The Examiner argues that processing limitations do not patentably distinguish a claimed product from a similar product in the prior art unless applicant shows the processing limitations inherently result in a materially different product. This rejection is respectfully but strenuously traversed for the reasons set forth in detail below.

First, the ranges of thicknesses for the individual layers disclosed in Bochow do not render the claimed ratios obvious. All of the examples of Bochow show thicknesses of unfilled layers higher than the thicknesses of the respective filled layers. Accordingly, a skilled artisan would not have been motivated to choose the proper ratios (with the unfilled layers being thinner than the filled layers) from the thicknesses of the layers listed in Bochow to achieve the critical ratios of the claimed invention.

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Bochow does not teach how much filler should b added, and Hattori in no way makes up for this deficiency. A skilled artisan would not have been motivated to combine Hattori with Bochow to achieve the claimed invention. Hattori seeks to maintain a proper balance of stiffness and thermo-formability while Bochow seeks a high degree of stiffness and limited thermo-formability. Accordingly, a skilled artisan seeking to achieve the limited thermo-formability of Bochow would not have looked to Hattori which tries to improve thermo-formability for the amount of filler to add to achieve this property.

Additionally, Bochow does not teach that the laminate may be formed on an FFS machine as claimed by the applicants. Examiner argues that the processing limitations do not patentably distinguish a claimed product from the prior art. However, Bochow discloses a different product than the claimed invention, as discussed above. Bochow achieves a product with limited thermo-formability which makes it a poor candidate for formation on an FFS machine while the claimed invention achieves excellent thermo-formability which makes it more suitable for complete and efficient formation on an FFS machine.

Even if prima facie obviousness could be shown based on any of the above-noted references or combinations of references, such prima facie obviousness is sufficiently overcome by applicant's improved and unexpected results of better thermo-forming characteristics resulting from the critical ratio of thickness of the filled layer to unfilled layers. The unsigned Section 132 Declaration of Bernig Walter, an employee of an affiliated company of the assignee of the present application, is submitted herewith and demonstrates that for the advantageous properties of the film, the paper-like appearance and the very good thermoforming properties evidenced by improved packaging speed, the ratio of the total thickness of the unfilled layers to the thickness of the filled layer is essential. Mr. Walter's signature could not be obtained by the response date as he was on Christmas vacation, but a signed Declaration will be submitted shortly after Mr. Walter's return for further consideration by the Examiner.

In particular, the comparison of Test IVa with Test IVb shows that the packaging speed for the films according to the claimed invention is higher than for films with a ratio of the total thickness of the unfilled layers to the thickness of filled layer outside of the inventive range. Further, the tests show that a film with a ratio of thickness of the unfilled layers to the thickness of the filled layer of 1:10 has paper-like features, but a narrow thermoforming temperature range

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(see Test IIa), and a film with a ratio of the total thickness of the unfilled layers to the thickness of the filled layer of 1:1.02 has a broad thermo-forming temperature range, but no paper-like features (see Test IIb).

In view of the forgoing amendments, remarks, enclosed ASTM 1238 and enclosed unsigned Declaration, applicants submit that the pending claims comply with the requirements of 35 U.S.C. § 112 and are patentably distinct from the prior art. Accordingly, reconsideration and withdrawal of the rejections and an early notice of allowance are respectfully requested.

Respectfully submitted,
ULRICH REINERS, *ET AL.*

January 7, 2001
(Date)

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Enclosures: Unsigned Declaration of Bernig Walter under 37 C.F.R. § 1.132
           Copy of ASTM 1238 from 1990
           Marked-Up Copy of Amended Claim 1

Attorney Docket No.
9784-3U2
(TH8002US/B)

Marked-Up Copy of Claim 1

1. (Amended) A [paper-like] multilayer barrier film <u>with an appearance and texture of paper</u> comprising a filled layer based on polypropylene and a plurality of unfilled layers, wherein the filled layer is filled with 40-75 weight % , based on the total weight of the filled layer, of an inorganic filler, forms one of the two surface layers of the film, and has a thickness of [³] 40 µm <u>to 400 µm</u> , the unfilled layers comprising at least a barrier layer and a sealing layer and optionally at least one adhesive layer, and the [relation] <u>ratio</u> of the total thickness of the unfilled layers to the thickness of the filled layer being from 1:8 to 1:1.2.

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANTS: Ulrich Reiners et al.

Application NO.:   09/851,460

FILED: 8 May, 2001

FOR: PAPER-LIKE AND THERMO-FORMABLE MULTILAYER
BARRIER FILM

DECLARATION UNDER 37 C.F.R. § 1.132

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

I, Bernig Walter, hereby declare as follows:

1.   I am a citizen of Germany, residing at Rottachbergweg 5
87549 Rettenberg

2.   I studied chemistry at the Fachhochschule of Aalen and received a degree in
the field chemical engineering in the year 1977.

3.   Since 1.4.1987 I have been employed as a project engineer/manager R&D in
the field of Research and Development films for deep draw applications and
shrink bags for shrink bag applications and I am still working in this field for
the company of Convenience Food Systems, Kempten, Germany, an
affiliated company of Convenience Food Systems B.V.

4.   The following tests were made under my supervision and control:

I. Films according to the US patent application 09/851,460

Ia. Test

A film with six layers is produced according to the blown film coextrusion procedure. The sequence of the layers is: AA`BCDE.

Layer A consists of: 53 weight-% of a calcium carbonate with an average particle size of 4,5 µm and 47 weight-% of a homopropylene with a melting index of 2,1 g/10 min.

Layer A` consists of: 100 weight-% of a LDPE with a melting index of 0,85 g/10 min and a density of 0,922 g/cm³.

Layer B consists of: 100 weight-% of an acid-modified ethylene methacrylate copolymer with a melting point of 108°C and a melting index of 3,0 /10 min and functions as adhesive layer.

Layer C consists of: 100 weight-% of an ethylene vinylalcohol copolymer with an ethylene percentage of 38 mol-% and a melting index of 5,5 g /10 min and functions as barrier layer.

Layer D consists of: 100 weight-% of an acid-modified polypropylene with a melting index of 3,5 g/10 min and functions as adhesive layer.

Layer E consists of: 87 weight-% of a polyethylene with a density of 0,9250 g/cm³ and a melting index of 2,0 g /10 min and 13 weight-% of a polybutylene with a melting index of 1,0 g /10 min and functions as sealing layer.

The thicknesses of the layers are:

Layer A	200 µm
Layer A`	10 µm
Layer B	3 µm
Layer C	4 µm
Layer D	3 µm
Layer E	20 µm.

The total thickness of the multilayer film is 240 µm. The ratio of the thickness of the unfilled layers to that of the filled layer is 1:5

Ib.   Test

In this text the layers of the multilayered film are composed as in Test Ia.

The thickness of each layer is:

Layer A	60 µm
Layer A`	15 µm
Layer B	3 µm
Layer C	4 µm
Layer D	3 µm
Layer E	23 µm.

The total thickness of the multilayer film is 109 µm. The ratio of the thickness of the unfilled layers to that of the filled layer is 1:1,27

II. Comparison films

IIa. Test

The film used has the same composition of the layers as described in Test Ia.

The thicknesses of the layers, however, are:

Layer A	350 µm
Layer A`	10 µm
Layer B	3 µm
Layer C	4 µm
Layer D	3 µm
Layer E	20 µm.

The total thickness of the multilayer film is 390 µm. The ratio of the thickness of the unfilled layers to that of the filled layer is 1:8,27

IIb. Test

The film used has the same composition of layers as in Test Ia.
The thicknesses of the layers, however, are as follows:

Layer A	60 µm
Layer A`	23 µm
Layer B	3 µm
Layer C	4 µm
Layer D	3 µm
Layer E	23 µm.

The total thickn ss of the multilayer film is 116 µm. The ratio of the thickness of the unfilled layers to that of the filled layer is 1:1,07.

The melting indices cited in the Tests were determined according to ASTM 1238.

III. Properties of the films according the Tests Ia to IIb

Film according to	range of thermo-forming (°C)	packaging speed (cycles per minute)*	appearance of the film
Test I a	125 to 145	12,5	paper-like
Test II a	145 to 156	10,4	paper-like
Test I b	115 to 145	12,8	paper-like
Test II b	115 to 145	12,8	plastic

* Determination of the packaging speed

Test IVa

Packaging articles (trays) made of the inventive film according to Test Ia are formed at 135°C for 15 min on a FFS machine. The process is interupted, the tray insert of the machine is changed and trays with another dimension made of the inventive film according to Ia are produced again at 135°C for 15 min. Then trays made of the inventive film according to Test Ib are formed at 135°C for 15 min. The process is interupted, the tray insert of the machine is changed and trays with another dimension made of the inventive film according to Test Ib are produced again at 135°C for 15 min.

Test IVb

Packaging articles (trays) made of the film according to Test IIa are formed at 145°C for 15 min on a FFS machine. Th process is interupted, the tray insert of th machin is changed and trays with another dimension made of the film according to Test IIa are produced again at 145°C for 15 min. Than trays made of the film according to

Test IIb are formed at 135°C for 15 min. The process is int rupted, the tray insert of the machine is changed and trays with another dimension made of the film according to Test IIb are produced again at 135°C for 15 min.

The packaging speed is determined as the number of produced packaging articles in one hour under the mentioned conditions.

V. Results

The Tests show that for the combination of advantageous properties of the film, as packaging material especially for the combination of the paper-like appearance and the very good thermoforming properties, the ratio of the total thickness of the unfilled layers to the thickness of the filled layer is essential. The comparison of Test IVa with Test IVb indicate that the packaging speed for the films according to the US patent application No. 09/851,460 is higher than for films with a ratio of the total thickness of the unfilled layers to the thickness of the filled layer outside the inventive range.

All statements made herein of my own knowledge are true, and all statements made on information and belief are believed to be true, and further, these statements were made with the knowledge that willful false statements and the like, so made, are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the patent application or any patent issued thereon.

_____   _____
(Dat)   (Bernig, Walter)

An American National Standard

ASTM Designation: D 1238 – 90b

Standard Test Method for
Flow Rates of Thermoplastics by Extrusion Plastometer[1]

This standard is issued under the fixed designation D 1238; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers measurement of the rate of extrusion of molten resins through a die of a specified length and diameter under prescribed conditions of temperature, load, and piston position in the barrel as the timed measurement is being made.

1.2 Procedure A is a manual cutoff operation based on time used for materials having flow rates that fall generally between 0.15 and 50 g/10 min. Procedure B is an automatically timed flow rate measurement used for materials having flows from 0.50 to 900 g/10 min. By both procedures, the piston travel is generally the same during the timed measurement; the piston foot is about 46 and 20.6 mm (1.81 and 0.81 in.) above the die. Comparable flow rates have been obtained by these procedures in interlaboratory round-robin measurements of several materials described in 12.1.

NOTE 1—Round-robin testing indicates this test method may be suitable at flow rates up to 1500 g/10 min if the timing clock resolves the elapsed time to the nearest 0.01 s.

1.3 The values stated in SI units are to be regarded as the standard. The values in parentheses are given for information only.

1.4 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in Note 9.

2. Referenced Documents

2.1 *ASTM Standards:*
D 618 Practice for Conditioning Plastics and Electrical Insulating Materials for Testing[2]
D 883 Terminology Relating to Plastics[2]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]
2.2 *American National Standards Institute Standard:*
B46.1 on Surface Texture[4]

3. Terminology

3.1 *General:*

3.1.1 For definition of some of the technical terms used in this standard refer to Terminology D 883.

4. Significance and Use

4.1 This test method is particularly useful for quality control tests on thermoplastics.

NOTE 2—Polymers having flow rates less than 0.15 or greater than 900 g/10 min may be tested by the procedures in this test method; however, precision data have not been developed.

4.2 This test method serves to indicate the uniformity of the flow rate of the polymer as made by an individual process and, in this case, may be indicative of uniformity of other properties. However, uniformity of flow rate among various polymers as made by various processes does not, in the absence of other tests, indicate uniformity of other properties.

4.3 The flow rate obtained with the extrusion plastometer is not a fundamental polymer property. It is an empirically defined parameter critically influenced by the physical properties and molecular structure of the polymer and the conditions of measurement. The rheological characteristics of polymer melts depend on a number of variables. Since the values of these variables occurring in this test may differ substantially from those in large-scale processes, test results may not correlate directly with processing behavior.

4.4 The flow rate of a material may be measured under any of the conditions listed for it in 8.2. Additional characterization of a material can be obtained if more than one condition is used. In case two conditions are employed, a Flow Rate Ratio (FRR) may be obtained by dividing the flow rate at one condition by the flow rate at the other condition.

5. Apparatus

5.1 *Plastometer:*

5.1.1 The apparatus shall be a dead-weight piston plastometer consisting of a thermostatically controlled heated steel cylinder with a die at the lower end and a weighted piston operating within the cylinder. The essential features of the plastometer, illustrated in Figs. 1 and 2, are described in 5.2 to 5.8.

5.1.2 Relatively minor changes in the design and arrangement of the component parts have been shown to cause differences in results between laboratories. It is important, therefore, for the best interlaboratory agreement that the design adhere closely to the description herein; otherwise, it should be determined that modifications do not influence the results.

5.2 *Cylinder*—The steel cylinder shall be 50.8 mm (2 in.) in diameter, 162 mm (6⅜ in.) in length with a smooth, straight hole 9.5504 ± 0.0076 mm (0.3760 ± 0.0003 in.) in diameter, displaced 4.8 mm (³⁄₁₆ in.) from the cylinder axis.
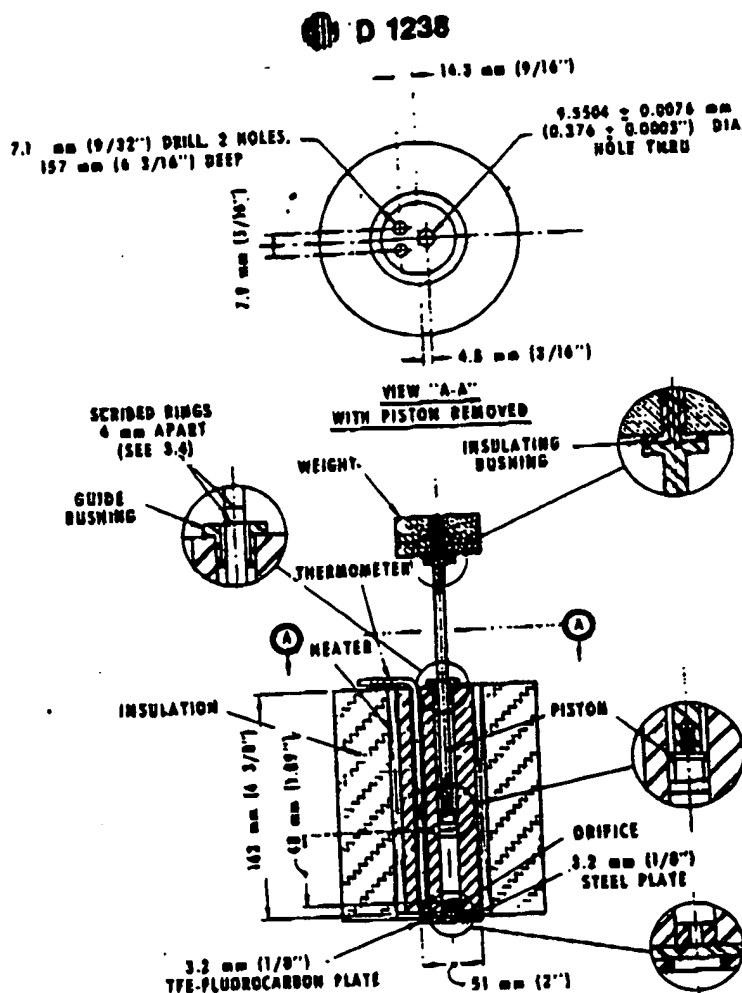
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FIG. 1 General Arrangement of Extrusion Plastometer

Wells for a thermal sensor (thermoregulator, thermistor, etc.) and thermometer shall be provided as shown in Fig. 1. A 3.2-mm (⅛-in.) plate shall be attached to the bottom of the cylinder to retain the die. A hole in this plate, centered under the die and countersunk from below, allows free passage of the extrudate. The cylinder may be supported by at least two 6.4-mm (¼-in.) high-strength screws at the top (radially positioned at right angles to the applied load) or by at least two 10-mm (⅜-in.) diameter rods screwed into the side of the cylinder for attaching to a vertical support. The essential dimensions of a satisfactory cylinder of this type are shown in Fig. 1 (Note 3). The cylinder bore should be finished by techniques known to produce approximately 12 rms or better in accordance with ANSI B46.1.

NOTE 3—Cylinders made of SAE 52100 steel heat-hardened to a Rockwell hardness, C scale, of 60 to 64 give good service when used at temperatures below 200°C.³ Cylinder liners of cobalt-chromium-tungsten alloy are also satisfactory to 300°C.

5.3 *Die*—The outside of the steel die shall be such diameter that it will fall freely to the bottom of the 9.5504-mm (0.3760-in.) diameter hole in the cylinder (Note 4). The die shall have a smooth straight bore 2.0955 ±

0.0051 mm (0.0825 ± 0.0002 in.) in diameter and shall t 8.000 ± 0.025 mm (0.315 ± 0.001 in.) in length. The bo: and its finish are critical. It shall have no visible drill or oth tool marks and no detectable eccentricity. The die bore sh be finished by techniques known to produce approximate 12 rms or better in accordance with ANSI B46.1.

NOTE 4—Die materials also found to be satisfactory are tungs carbide,⁶ synthetic sapphire, and cobalt-chromium-tungsten alloy.³

5.4 *Piston:*
5.4.1 The piston shall be made of steel with an insulati bushing at the top as a barrier to heat transfer from piston to the weight. The land of the piston shall be 9.4742 0.0076 mm (0.3730 ± 0.0003 in.) in diameter and 6.35 0.13 mm (0.250 ± 0.005 in.) in length. The piston des may incorporate means for land replacement, for exam} having threads and flats immediately above the land. Ab the land, the piston shall be relieved to 8.890 ± 0.025 n (0.350 ± 0.001 in.) in diameter (Note 5). The finish of piston foot shall be 12 rms in accordance with ANSI B4(

NOTE 5—To improve standardization it is preferable that the p be sleeved at the top and be relieved as described in 5.4.2.
NOTE 6—Pistons of SAE 52100 steel with the bottom 25 mm (1

³ Haynes Stellite 6B. manufactured by the Haynes Stellite Co., Kokomo, IN. has been found satisfactory for this purpose.

⁶ Talide C-99, manufactured by the Metal Carbides Corp., Youngstown, has been found satisfactory for this purpose.
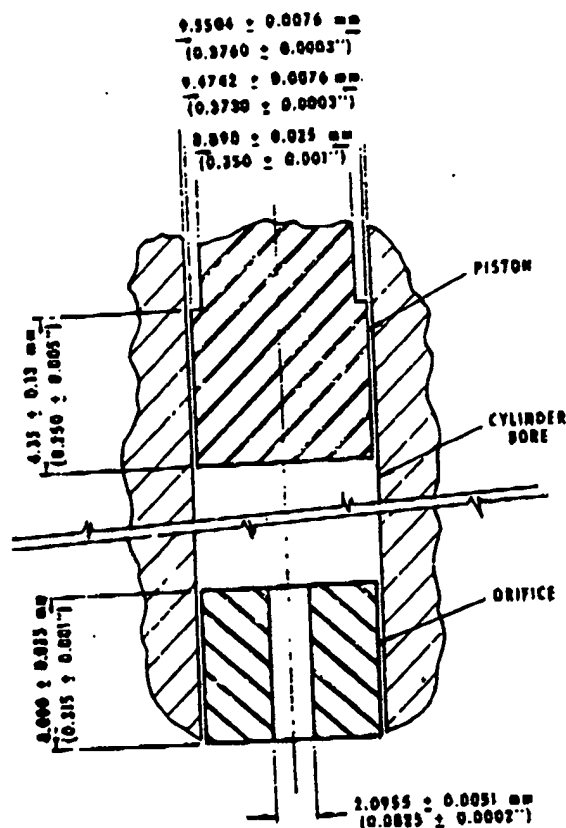
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FIG. 2 Details of Extrusion Plastometer

including the foot, hardened to a Rockwell hardness, C scale, of 55 to 59 have been found to give good service when used at temperatures below 200°C.

5.4.2 A solid piston relieved to 7.900 ± 0.025 mm (0.311 ± 0.001 in.) in diameter above the land with a loose-fitting metal guiding sleeve at the top of the cylinder is desirable for use with the 21.6 kg load. If wear or corrosion is a problem, the piston should be of stainless steel and equipped with a detachable foot for ease of replacement.

5.4.3 For Procedure A the piston shall be scribed with two reference marks 4 mm apart in such fashion that when the lower mark coincides with the top of the cylinder or other suitable reference point, the bottom of the piston is 48 mm (1.89 in.) above the top of the die (see Fig. 1).

5.4.4 The combined weight of piston and load shall be within a tolerance of ±0.5 % of the selected load.

5.5 *Heater:*

5.5.1 Provision shall be made for heating the apparatus so that the temperature of the material can be maintained within ±0.2°C of the desired temperature during the test. At temperatures higher than 200°C, this degree of temperature control may be more difficult to obtain. The temperature specified shall be the equilibrium temperature of the material 12.7 mm (½ in.) above the die.

5.5.2 Calibrate the temperature-indicating device by means of a light-gage probe-type thermocouple or a platinum-resistance temperature sensor having a short sensing

length.[7] The thermocouple should be encased in a metallic sheath having a diameter of approximately 1.6 mm (¹⁄₁₆ in.) with its hot juncti n grounded to the end of the sheath. Insert the temperature sensor into the melt from the top of the cylinder so that it is 12.7 mm (½ in.) above the upper face of the die. The temperature sensors shall be used with a potentiometer having a sensitivity of at least 0.005 mV, or a temperature readout having a sensitivity of at least 0.1°C. It is desirable to calibrate the thermocouple or platinum-resistance temperature sensor and readout equipment with reference to a platinum resistance temperature thermometer. Comparison of the temperature sensor and readout of a calibrated thermometer similar to that described in 4.7, using the thermometer well containing heat-transfer medium, has also been found useful.

5.5.3 Heat shall be supplied by an electric band heater which covers the entire length of the cylinder. The heater may be a single- or multi-element heater, depending upon manufacturer's control means. The heater plus control system must be capable of maintaining the set points within the required ±0.2°C. If the heater contains two 100-W concentric elements, as close to 90 % as feasible of the power required to maintain the cylinder at the specified temperature should be supplied continuously by the outer of the two heating elements. Six tenths of this amount of power shall be applied intermittently by the inner element as required to maintain the specified temperature (Note 7). The cylinder with heater shall be lagged with 38 mm (1.5 in.) of foamed-glass insulation.[8] A TFE-fluorocarbon plate 3.2 mm (⅛ in.) in thickness shall be attached to the bottom of the cylinder to minimize heat loss at this point.

NOTE 7—Experience has shown that the correct adjustment of the power to the heaters is important in order that all instruments possess identical thermal characteristics. A convenient method for adjustment is obtained by measuring the voltage required to maintain the cylinder at the specified temperature (±1°C) using only the constant heater. Then the voltage to the constant heater is readjusted to the voltage obtained from the calculation:

$$E_c = \sqrt{0.9e^2} = 0.95e$$

where:

E_c = voltage required to maintain approximately 90 % of the power required to keep the cylinder at the specified temperature, and

e = voltage required to maintain the power required to keep the cylinder at the specified temperature.

The intermittent heater voltage is then obtained by the calculation:

$$E_i = \sqrt{0.6e^2} = 0.78e$$

where:

E_i = voltage specified for the intermittent heater, and

e = same as above.

Measurement of the voltages is imperative because variable autotransformer settings do not necessarily represent voltages.

5.6 *Temperature Controller*—The type of controller and sensor must be capable of meeting the required control tolerance specified in 5.5.1.

[7] Round-robin data showing flow rate and corresponding temperature profile of the melt obtained using probe-type thermocouples and platinum resistance temperature sensors can be obtained from ASTM Headquarters. Request RR:D20-1094.

[8] "Foamglas" 5.4-cm (2⅛-in.) diameter IW pipe insulation as manufactured by the Pittsburgh Corning Corp., Pittsburgh, PA, is recommended for this purpose.

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NOTE 8—Wood's metal,[9] as a heat transfer medium, may be used with the indicating or control elements if required for accuracy or stability by a particular control system. Care sh uld be taken that the Wood's metal is suitable for the selected temperature. Elements that could sustain damage upon cooling with Wood's metal should be removed prior to the cool down. Silicon oil, as a heat transfer medium, should be avoided based upon possible contamination of the extrusion plastometer cylinder and its effect on flow rate.

5.7 *Thermometer*—Thermometers having a range of 4°C graduated in 0.2°C divisions may be used to indicate temperature. The temperature at this point may not necessarily be the temperature of the material 12.7 mm (½ in.) above the die. The thermometer may be used to monitor indirectly the temperature of the material 12.7 mm (½ in.) above the die and may be calibrated by reference to a thermocouple inserted in the material 12.7 mm (½ in.) above the die. See 5.5.2 for description of thermocouple and method for measuring temperature.

NOTE 9—Warning: Caution should be observed with the use of a mercury-filled thermometer. Mercury vaporization occurs if the thermometer is broken. Mercury thermometers are not to be used at or above the boiling point of mercury, which is 357°C.

5.8 *Level*—Provision shall be made for vertical alignment of the bore of the extrusion plastometer. This is necessary to minimize subtractive loads resulting from rubbing or friction between the piston tip and sidewall. Means of alignment are discussed in Appendix X1.

5.9 *Accessory Equipment*—Necessary accessories include equipment for charging samples to the cylinder, a funnel, a die plug, a tool for cutting off the extruded sample, a timer or stop watch, cleaning equipment, and a balance accurate to ±0.001 g.

NOTE 10—Satisfactory operation of the apparatus for polyethylenes can be ascertained by making measurements on NIST Standard Reference Materials (SRM) Nos. 1475 and 1476.[10] These SRM polyethylenes are, respectively, a linear polyethylene having a flow rate of 2.07 g/10 min at test condition 190/0.325 and a branched polyethylene having a flow rate of 1.19 g/10 min at test condition 190/2.16 (melt index).

6. Test Specimen

6.1 The test specimen may be in any form that can be introduced into the bore of the cylinder, for example, powder, granules, strips of film, or molded slugs. It may be desirable to preform or pelletize a powder.

7. Conditioning

7.1 Many thermoplastic materials do not require conditioning prior to testing. Materials which contain volatile components, are chemically reactive, or have other special characteristics most probably require appropriate conditioning procedures. Moisture not only affects reproducibility of flow rate measurement but, in some types of materials, degradation is accelerated by moisture at the high temperatures used in testing. If conditioning is necessary, see the applicable material specification and Practice D 618.

[9] Wood's Metal or an aluminum powder may be used. Wood's metal is available from Belmont Smelting and Refining Works, Inc., 320 Belmont Ave., Brooklyn, NY 11207.

[10] These standard polyethylenes are available from the National Institute of Standards and Technology, Office of Standard Reference Materials, Washington, DC 20234.

8. Procedural Conditions

8.1 Standard conditions of test are given in Table 1. The previously used alphabetized system for designating test conditions is shown in Table 1 f r reference only. Test conditions shall be shown as: Condition ___ / ___, where the temperature in degrees Celsius is shown first, followed by the weight in kilograms. For example: Condition 190/2.16 (formerly Condition E).

8.2 The following conditions have been found satisfactory for the material listed:

Material	Condition	
Acetals (copolymer and homopolymer)	190/2.16	190/1.05
Acrylics	230/1.2	230/3.8
Acrylonitrile-butadiene-styrene	200/5.0	230/3.8
Cellulose esters	190/0.325	190/2.16
	190/21.60	210/2.16
Nylon	275/0.325	235/1.0
	235/2.16	235/5.0
	275/5.0	
Polychlorotrifluorethylene	265/12.5	
Polyethylene	125/0.325	125/2.16
	190/0.325	190/2.16
	190/21.60	190/10
	310/12.5	
Polycarbonate	300/1.2	
Polypropylene	230/2.16	
Polystyrene	200/5.0	230/1.2
	230/3.8	190/5.0
Polyterephthalate	250/2.16	210/2.16
	285/2.16	
Poly(vinyl acetal)	150/2.16	
Poly(phenylene sulfide)	315/5.0	
Styrene acrylonitrile	220/10	230/10
	230/3.8	
Styrenic Thermoplastic Elastomer	190/2.16	200/5.0
Thermoplastic Elastomer-Ether-Ester	190/2.16	220/2.16
	230/2.16	240/2.16
		250/2.16

8.3 If more than one condition is used and the material is polyethylene, the determination of Flow Rate Ratio (FRR) has been found to be useful. FRR is a dimensionless number derived by dividing the flow rate at Condition 190/10 by the flow rate at Condition 190/2.16.

NOTE 11—When determining such a ratio of flow rates for a material at the same temperature under different loads, it has been found that precision is maximized when one operator uses one Procedure (A or B), the same plastometer, and the same die for both measurements (the die need not be removed from the plastometer between the two determinations).

NOTE 12—Moisture sensitivity can significantly influence flow rate results for some materials. Appropriate material specifications should be referenced for specific pre-drying or sample handling instructions.

9. Procedure A—Manual Operation

9.1 Select conditions of temperature and load from Table 1 in accordance with material specifications such that flow rates will fall between 0.15 to 50 g/10 min.

9.2 Ensure that the bore of the extrusion plastometer is properly aligned in the vertical direction. (See Appendix X1).

9.3 The apparatus shall be clean. The parts are more easily cleaned while they are hot. The temperature of the cylinder with piston and die in place shall have been at the test temperature for at least 15 min before a test is begun. When the equipment is used repetitiously, it should not be necessary to heat the piston and die for 15 min. See Note 7 for procedure for adjusting temperature. Take care that cleaning or previous use shall not have changed the dimensions. Make frequent checks to determine whether the die

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TABLE 1 Standard Test Conditions, Temperature, and Load

Condition Obsolete-For-reference only	Standard Designation	Temperature, °C	Total Load Including Piston, kg	Approximate Pressure kPa	psi
A	125/0.325	125	0.325	44.8	6.5
B	125/2.16	125	2.16	298.2	43.25
C	150/2.16	150	2.16	298.2	43.25
D	190/0.325	190	0.325	44.8	6.5
E	190/2.16	190	2.16	298.2	43.25
F	190/21.60	190	21.60	2982.2	432.5
G	200/5.0	200	5.0	689.5	100.0
H	230/1.2	230	1.2	165.4	24.0
I	230/3.8	230	3.8	524.0	76.0
J	265/12.5	265	12.5	1723.7	250.0
K	275/0.325	275	0.325	44.8	6.5
L	230/2.16*	230	2.16	298.2	43.25
M	190/1.05	190	1.05	144.7	21.0
N	190/10.0	190	10.0	1379.0	200.0
O	300/1.2	300	1.2	165.4	24.0
P	190/5.0	190	5.0	689.5	100.0
Q	235/1.0	235	1.0	138.2	20.05
R	235/2.16	235	2.16	298.2	43.25
S	235/5.0	235	5.0	689.5	100.0
T	250/2.16	250	2.16	298.2	43.25
U	310/12.5	310	12.5	1723.7	250.0
V	210/2.16	210	2.16	298.2	43.25
W	285/2.16	285	2.16	298.2	100.0
X	315/5.0	315	5.0	689.5	

diameter is within the tolerances given in 5.3.

9.4 Remove the piston and place it on an insulated surface. Charge the cylinder within 1 min with a weighed portion of the sample according to the expected flow rate, as given in Table 2. Place the weighted piston in position, and start timing for a 6 to 8-min preheat.

NOTE 12—Experience has shown that for the best reproducibility the piston should operate within the same part of the cylinder for each measurement. The piston is scribed so the starting point for each extrusion is roughly the same. Some excess of material over the minimum required for the actual flow measurement portion of the test is provided by the charging weights shown in Table 2. This is necessary to achieve a void-free extrudate and flow equilibrium before start of rate measurements. For the lower flow rate materials, it may be necessary to manually force some of the excess resin out of the cylinder to obtain proper scribe mark position within the specified preheat time.

NOTE 13—There may be cases where 6 to 8 min of preheat time may not be sufficient. Six minutes is a minimum preheat time. Longer preheat periods are permissible when they can be shown to be necessary. If longer preheat times are used, the report shall so indicate. Care may be necessary to ensure that the sample so tested is adequately protected against changes in polymer structure, usually by appropriate antioxidant addition.

NOTE 14—It is frequently helpful to take interim cuts of the extrudate at uniform time intervals during the specified extrusion time. Weights of these individual cuts give an indication of the presence of bubbles which may be masked due to their size or to opacity of the sample. This technique is particularly helpful in the case of highly pigmented materials. Forcing out some of the resin manually during the preheat period often eliminates bubbles in the test extrudate.

9.5 For flow rates of 0.15 to 10 g/10 min, if the weighted piston alone does not purge sufficient material during the preheat time to obtain piston positioning as required in 9.6, manually purge the estimated excess prior to 4 min. The amount of purge must be regulated so subsequent free travel of the weighted piston will position the scribe marks at the reference start position between the 6th and 8th min (9.6). Manual purging in this manner is permissible only when it is

known that it will not affect results. Otherwise, purge sooner, or use a lower charge weight.

9.6 For flow rates of greater than 10 to 50 g/10 min, the loss of weighed sample as shown in Table 2 could be unduly large by proceeding as in 9.4 without modification. In this case, during the first 6 min of preheat time, either a piston support or a die plug may be used.

9.6.1 A piston support should be of such length that the lower scribe mark of the supported piston will be 25 mm (1 in.) above the top of the cylinder or other reference point (5.4.3). The support may be a piece of wooden dowel rod or the like of appropriate length beneath the weight, to support the weight and thus the piston in the desired position. The

TABLE 2 Standard Test Conditions, Sample Weight,[A] and Testing Time—Procedure A[B]

Flow Range, g/10 min	Suggested Weight of Sample in Cylinder, g	Time Interval, min	Factor for Obtaining Flow Rate in g/10 min
0.15 to 1.0	2.5 to 3.0	6.00	1.67
>1.0 to 3.5	3.0 to 5.0	3.00	3.33
>3.5 to 10	5.0 to 8.0	1.00	10.00
>10 to 25	4.0 to 8.0	0.50	20.00
>25 to 50	4.0 to 8.0	0.25	40.00

[A] This is a suggested weight for materials with melt densities of about 0.7 g/cm³. Correspondingly, greater quantities are suggested for materials of greater melt densities. Density of the molten resin (without filler) may be obtained using the procedure described by Terry, B. W., and Yang, K., "A New Method for Determining Melt Density as a Function of Pressure and Temperature," SPE Journal, SPEJA, Vol 20, No. 6, June 1964, p. 540 or the procedure described by Zoller, Paul, "The Pressure-Volume-Temperature Properties of Polyolefins," Journal of Applied Polymer Science, Vol 23, 1979, p. 1051. It may also be obtained from the weight of an extruded known volume of resin at the desired temperature. For example, 25.4 mm (1 in.) of piston movement extrudes 1.804 cm³ of resin. An estimate of the density of the material can be calculated from the following equation:

Resin density at test temperature = W/1.804

where W = weight of extruded resin.

[B] See 9.11.

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amount of weighed sample should be such that at least 1 g of material is purged through the die during the melting period, as the weight descends to the point wherein it is supported by the stop. Remove the support after 6 min of preheat.

9.6.2 When a die plug is used, the plug is inserted into the die before the weighed sample is charged. The die plug may be a brass pin nominally 2.05 mm (0.081 in.) in diameter and 25 mm (1 in.) long, projecting 8 mm (0.315 in.) from the end f a wooden dowel rod or other poor heat conductor. The length of the rod, conveniently, is such that one of the spare weights will just slip beneath it to hold it in place after the plug is inserted into the die from below. When a plug is used, adjust the amount of charge to provide a melted volume that will support the piston so that the lower scribe mark is approximately 25 mm (1 in.) above the top of the cylinder or other reference point. Remove the plug after 6 min of preheat.

NOTE 15—Care should be taken in removing the plug to avoid contact with molten extrudate.

9.7 For all tests, start collecting a timed extrudate when requirements for piston position are met, provided this is between 6 and 8 min from charging (Note 13); otherwise, discard the charge and readjust the charge weight or manual position during preheat. Requirements are that the top scribed mark on the piston be visible above the cylinder or index and that the lower scribed mark be in the cylinder or below the index. As the lower scribed mark approaches the top of the cylinder or index, reset the timer to zero, then simultaneously start the timer and make the cut-off when position requirements are met. Collect the next extruded portion exactly according to the time interval given in Table 2 (Note 14). If the extrudate contains visible bubbles, discard the complete charge and begin the test again.

9.8 Discharge the remainder of the specimen and push the die out through the top of the cylinder. Swab out the cylinder with cloth patches after the manner of cleaning a pistol barrel. The die may be cleaned by dissolving the residue in a solvent. A better method is pyrolytic decomposition of the residue in a nitrogen atmosphere. Place the die in a tubular combustion furnace or other device for heating to 550 ± 10°C and clean with a small nitrogen purge through the die. This method is preferable to flame or solvent cleaning, being faster than solvent cleaning and less detrimental to the die than an open flame. In certain cases where materials of a given class having similar flow characteristics are being tested consecutively, interim die cleaning may be unnecessary. In such cases, however, the effect of cleaning upon flow rate determination must be shown to be negligible if this step is avoided.

9.9 Weigh the extrudate to the nearest 1 mg when cool.

9.10 Multiply the weight by the factor shown in Table 2 to obtain the flow rate in grams per 10 min.

NOTE 16—Frequently, errors in test technique, apparatus geometry, or test conditions, which defy all but the most careful scrutiny exist, causing discrepancy in flow rate determinations. The existence of such errors is readily determined by periodically measuring a reference sample of known flow rate. The flow rate value and range to be tolerated can be determined using a statistically correct test program composed of multiple determinations with various instruments. Standard samples of polyethylene, linear branched, are available from the National Institute of Standards and Technology.

9.11 In case a specimen has a flow rate at the borderline of the ranges in Table 2 and slightly different values are obtained at different time intervals, the referee value shall be obtained at the longer time interval.

10. Procedure B—Automatically Timed Flow Rate Measurement

10.1 *Apparatus:*

10.1.1 Extrusion plastometer and auxiliary equipment are detailed in Section 4 and below.

10.1.2 A timing device shall electrically or mechanically time piston movement within the specified travel range. The requirements of the system are as follows:

10.1.2.1 Sense and indicate time within ±0.1 s (See Note 1).

10.1.2.2 Measure piston travel within ±0.025 mm (±0.001 in.) for use in the flow rate calculations.

10.1.2.3 Any effects on the applied load must be included in the allowable tolerance given in 5.4.1.

10.1.2.4 It should be preset or be settable for measuring piston travel of 6.35 ± 0.25 mm (0.250 ± 0.010 in.) for flow rates up to 10 g/10 min.

10.1.2.5 It should be preset or be adjustable for measuring piston travel of 25.4 ± 0.25 mm (1.000 ± 0.010 in.) for flow rates greater than 10 g/10 min.

10.2 *Procedure:*

10.2.1 To ensure high interlaboratory reproducibility, it is important that the timing device operates within a fixed portion of the cylinder. This is defined as the portion of the cylinder between 46 ± 2 mm (1.81 ± 0.079 in.) and 20.6 ± 2 mm (0.81 ± 0.079 in.) above the top of the die.

10.2.2 Check die, cylinder, and position dimensions for conformance to 5.2 through 5.4 and Figs. 1 and 2.

10.2.3 Refer to Table 1 for selection of conditions of temperature and load in accordance with the material specification.

10.2.4 Set the temperature of the plastometer as required for the material being tested and with voltage applied to the heaters as detailed in Note 7.

10.2.5 Ensure that the bore of the extrusion plastometer is properly aligned in the vertical direction. (See Appendix X1.).

10.2.6 Clean the apparatus thoroughly prior to each test (see 9.8). Position the piston and die in the cylinder and seat firmly on the base plate. Maintain the temperature for at least 15 min before beginning a test. When the equipment is used repetitiously, it should not be necessary to heat the piston and die for 15 min.

10.2.7 For flow rates of 0.50 to 10 g/10 min charge the cylinder with a weighed portion of the sample depending on the expected flow rate of the material as shown in Table 3. Place the weighted piston in place, and start the stop-watch or preheat timer. For samples whose flow rate is greater than 10 g/10 min, insert a plug into the die prior to charging the

TABLE 3 Suggested Sample Charge Weight—Procedure B

Flow Rate, g/10 min	Suggested Weight of Sample, g
0.5–3.5	2.0–4.0
>3.5–300	4.0–8.0

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weighed sample as shown in Table 3 or use a piston support (see 9.6.1).

10.2.8 If it is necessary to force out excess resin manually so as to adjust the position of the piston in the cylinder, do this before 4 min of preheat. For expected flow rates greater than 10 g/10 min remove the piston stop or remove the plug at 6 min. Small deviation from the expected flow rate is permissible.

10.2.9 Upon subsequent free travel, the timer must be actuated between the sixth and eighth minute of total preheat time, otherwise discard the charge and readjust the charge weight.

10.2.10 Observe the extrudate for voids during the flow rate measurement time. Also note condition of the extrudate leaving the orifice for any indication of material leakage around the sides of the die.

10.2.11 Record the time to the nearest 0.1 s for the piston to complete the calibrated distance of travel. Discard any runs with voids occurring in the timed portion or leakage of extrudate around the die.

10.2.12 Discharge any remaining resin and clean die and cylinder as detailed in 8.8.

11. Calculation (Procedure B)

11.1 Calculate the flow rate in grams per 10 min as follows (Note 16):

$$\text{Flow rate} = (427 \times L \times d)/t$$

where:
L = length of calibrated piston travel, cm,
d = density of resin at test temperature, g/cm^3 (see reference under Table 2),
t = time of piston travel for length L, s, and
427 = mean of areas of piston and cylinder × 600.

NOTE 17—Factors that may be substituted in the following equation are given for some materials in Table 4.

$$\text{Flow rate, g/10 min} = F/t$$

where:
F = factor from Table 4, and
t = time of piston travel for length L, s.

NOTE 18—It has been found, however, that agreement between Procedures A and B may be optimized if an average melt density for a particular type material is determined with the actual equipment used and that value substituted into the equation given in 11.1.

12. Report

12.1 Report the following information:

12.1.1 Statement indicating the nature and physical form of the material charged to the cylinder.

12.1.2 Temperature and load at which the test is run shall be reported. The results and test conditions can be referred to as FR-condition, where the standard designation for the condition from Table 1 is shown (for example: FR-190/2.16).

NOTE 19—It has become customary to refer to the flow rate of polyethylene as "melt index" when obtained under Condition 190/2.16. However, for all other materials the use of melt index or any term other than "flow rate" is discouraged, regardless of the condition used.

12.1.3 Flow rate reported as the rate f extrusi n in grams per 10 min.

12.1.4 Procedure used (A or B).

12.1.5 Any unusual behavior of the test specimen such as

TABLE 4 Factors for Calculation of Flow Rate

Material (Unpigmented)	Temperature, °C	Piston Travel, L, cm (in.)	Factor for Calculation of Flow Rate, F [a]
Polyethylene	190	2.54 (1)	828
Polyethylene	190	0.635 (0.25)	207
Polypropylene	230	2.54 (1)	801
Polypropylene	230	0.635 (0.25)	200

[a] Factors calculated using melt-density values of 0.7636 g/cm^3 for polyethylene and 0.7386 g/cm^3 for polypropylene, as expressed in article by Zoller, Paul, "The Pressure-Volume-Temperature Properties of Polyolefins," Journal of Applied Polymer Science, Vol 23, 1979, p. 1051. The base densities at 23°C for which the melt densities are reported were 0.917 g/cm^3 and 0.906 g/cm^3 for annealed low-density polyethylene and polypropylene homopolymer.

discoloration, sticking, extrudate surface irregularity or roughness, etc.

12.1.6 Details of conditioning, if any.

13. Precision and Bias (Procedures A and B)

13.1 *Precision:*

13.1.1 Tables 5 and 6 are based on a round robin[11] conducted in 1986 and 1987, involving polypropylene, polyethylene, polystyrene, polycarbonate and acrylic materials. The number of participating laboratories is shown for each material. Each laboratory tested two specimens for each material on three different days. The analysis in Practice E 691 is based on a test result being the average of two specimens.

13.1.2 Table 7 is based on a round robin[12] conducted in 1980 using Procedure B. Four polypropylene samples having flow rates from 250 to 1500 were tested in 9 laboratories.

NOTE 20: Caution—The following explanations of I_r and I_R (13.1.3 through 13.1.5) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Tables 5 to 7 should not be vigorously applied to acceptance or rejection of material since those data are specific to the round robin and may not be representative of other lots, conditions, materials or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials. The principles of 13.1.3 through 13.1.6 would then be valid for such data.

13.1.3 *Concept of I_r and I_R*—Relevant if S_r and S_R have been calculated from a large enough body of data, and if test results are averages obtained from testing two specimens.

13.1.4 *Repeatability, I_r*—In comparing two test results for the same material, obtained by the same operator using the same equipment on the same day, the two test results should be judged not equivalent if they differ by more than the I_r value for that material.

13.1.5 *Reproducibility, I_R*—In comparing two test results for the same material, obtained by different operators using different equipment on different days, the two test results should be judged not equivalent if they differ by more than the I_R value for that material.

13.1.6 Any judgment in accordance with 13.1.3 and 13.1.5 would have an approximate 95 % (0.95) probability f being correct.

13.2 *Bias*—There are no recognized standards by which to estimate bias f this test method.

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[11] Supporting data are available from ASTM Headquarters. Request RR: D-20-1104.
[12] Supporting data are available from ASTM Headquarters. Request RR: D-20-1124.

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TABLE 5 Precision, Procedure A (values in g/10 min)

Material	Condition	Average	S_r^A	S_R^B	I_r^C	I_R^D	No. of Laboratories
Polyethylene	190/2.16	0.27	0.006	0.022	0.023	0.063	9
Polyethylene	190/2.16	0.40	0.012	0.038	0.035	0.108	9
Polyethylene	190/2.16	2.04	0.026	0.079	0.073	0.224	9
Polyethylene	190/2.16	44.1	0.919	1.232	2.560	3.486	7
Polypropylene	230/2.16	2.23	0.106	0.226	0.299	0.639	9
Polypropylene	230/2.16	7.09	0.222	0.471	0.627	1.331	9
Polypropylene	230/2.16	32.8	0.581	1.061	1.644	2.974	6
Polystyrene	200/5	1.67	0.024	0.122	0.068	0.344	6
Polystyrene	200/5	8.82	0.190	0.667	0.538	1.886	6
Polystyrene	200/5	13.3	0.305	0.925	0.864	2.617	4
Polycarbonate	300/1.2	2.41	0.076	0.115	0.215	0.326	4
Polycarbonate	300/1.2	10.5	0.429	0.647	1.215	1.830	4
Polycarbonate	300/1.2	16.2	0.155	1.109	0.438	3.140	3
Acrylic	230/3.8	2.59	0.051	0.061	0.145	0.145	

^{A} S_r = within-laboratory standard deviation of the average.
^{B} S_R = between-laboratories standard deviation of the average.
^{C} I_r = 2.83 S_r, and
^{D} I_R = 2.83 S_R.

TABLE 6 Precision, Procedure B (values in g/10 min)

Material	Condition	Average	S_r^A	S_R^B	I_r^C	I_R^D	No. of Laboratories
Polyethylene	190/2.16	0.27	0.009	0.014	0.026	0.039	8
Polyethylene	190/2.16	0.40	0.016	0.027	0.045	0.076	8
Polyethylene	190/2.16	2.04	0.040	0.094	0.112	0.266	9
Polyethylene	190/2.16	43.7	0.997	1.924	2.819	5.443	8
Polypropylene	230/2.16	2.25	0.052	0.214	0.1466	0.604	8
Polypropylene	230/2.16	7.16	0.143	0.589	0.4051	1.666	8
Polypropylene	230/2.16	32.8	0.693	0.946	1.959	2.672	8
Polystyrene	200/5	1.65	0.037	0.166	0.106	0.470	4
Polystyrene	200/5	8.39	0.144	0.423	0.406	1.197	4
Polystyrene	200/5	13.0	0.108	0.387	0.306	1.097	4

^{A} S_r = within-laboratory standard deviation of the average.
^{B} S_R = between-laboratories standard deviation of the average.
^{C} I_r = 2.83 S_r, and
^{D} I_R = 2.83 S_R.

TABLE 7 Precision, Procedure B (values in g/10 min)

Material	Condition	Average	S_r^A	S_R^B	I_r^C	I_R^D
Polypropylene	230/2.16	245	13.2	16.6	37.4	46.9
Polypropylene	230/2.16	462	31.8	40.0	89.9	113
Polypropylene	230/2.16	837	20.9	58.6	59.1	166
Polypropylene	230/2.16	1603	129	243	365	688

^{A} S_r = within-laboratory standard deviation of the average.
^{B} S_R = between-laboratories standard deviation of the average.
^{C} I_r = 2.83 S_r, and
^{D} I_R = 2.83 S_R.

APPENDIX

(Nonmandatory Information)

X1. EXTRUSION PLASTOMETER BORE ALIGNMENT

X1.1 A fixture consisting of a circular level mounted on a shaft having two bearing points 9.47 + 0.00 − 0.0076 mm (0.373 + 0.00 − 0.0003 in.) in diameter that can be inserted into the bore has been found suitable. A circular level that can be rigidly mounted on the piston rod for insertion into the bore may also be satisfactory. A circular level having a sensitivity of 20 minutes per 2.5 mm (0.1 in.) has been found satisfactory. Other alignment techniques that give comparable alignment sensitivity would be considered satisfactory.

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REFERENCES

(1) "Polyethylene Insulaton and Sheathing for Electrical Cables." *Government Department Electrical Specification No. 27.* Great Britain. 1950.

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(3) Dexter, F. D., "Plasticity Grading of Fluorothenes," *Modern Plastics,* MOPLA. Vol 30, No. 8, 1953, p. 125.

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(5) Rudin, A., and Schreiber, H. P., "Factors in Melt Indexing of Polyolefins." *SPE Journal,* SPEJA. Vol 20, No. 6, 1964, p. 533.